# PATENT SPECIFICATION (11) 1232929

1232929

NO DRAWINGS

(21) Application No. 20952/69 (22) Filed 24 April 1969

(31) Convention Application No. 726 614 (32) Filed 3 May 1968 in

(33) United States of America (US)

(45) Complete Specification published 26 May 1971

(51) International Classification A 23 g 5/00

(52) Index at acceptance A2B 16



## (54) AERATED SOFT-SERVE COMPOSITIONS


(71) We, E. I. DU PONT DE NEMOURS AND COMPANY, a corporation organised and existing under the laws of the State of Delaware, United States of America, of Wilmington, State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—


This invention relates to soft-serve products which contain selected non-toxic chlorofluorohydrocarbon aerating agents dispersed therein.

In this specification, the expression "soft-serve product" means a frozen or semi-frozen product obtained by subjecting a liquid mix comprising milk fat, vegetable fat or animal fat, to a temperature between −17.7°C and −1.1°C, the mix thickening and congealing without freezing to a hard solid when subjected to a temperature between −17.7°C and −1.1°C. Examples of soft-serve products are soft ice cream, frozen and semi-frozen milk shakes and sherbets, and frozen custard.

Unfrozen, liquid soft-serve mix is ordinarily aerated, usually with air, during the freezing step so that the volume of the finished product exceeds the volume of the unfrozen mix. The increase in volume is known as "overrun". It is advantageous to the operator of soft ice cream apparatus to maximize the amount of overrun without losing the saleable qualities of the soft serve, such as texture and taste.

In the usual soft-serve preparation, the liquid mix becomes stiff due to its congealing during freezing. This causes a decrease in the amount of overrun because the rate of movement of the beater element, which mixes the soft serve with the aerating gas, is slowed. Moreover, when the beaten, congealed mix is allowed to stand, due to non-use, in the freezing zone, the amount of overrun decreases due to breakdown of the aerating gas cells. These problems can be overcome by uniformly aerating the soft-serve mix under pressure to a preselected degree of overrun before injection into the freezing zone, and then freezing under pressure to a desired degree of congelation without any active beating.

However, with the usual aerating gases, such as air or nitrous oxide, overruns of at most only 80% could be obtained without degeneration of the product. Moreover, even with overruns less than 80 per cent, the product appeared soft and was wet and runny. These factors created drainage or leakage problems for the operator of the apparatus; and the rapid melting of the soft-serve product upon exposure to ambient temperatures created problems for the consumer.

We have now found that soft-serve products can be obtained with overruns of up to 110% or more while maintaining a flavourable, firm, non-drip and non-grainy texture, by adding to the soft-serve mix chloropentafluoroethane, chlorotrifluoromethane, mixtures of both, or mixtures of each with nitrous oxide.

According to the invention, therefore, we provide an aerated soft-serve product (as herein defined) having intimately dispersed therein, as aerating agent, chloropentafluoroethane, chlorotrifluoromethane, a mixture of chloropentafluoroethane and chlorotrifluoromethane, a mixture of chloropentafluoroethane and nitrous oxide, or a mixture of chlorotrifluoromethane and nitrous oxide.

The aerated soft-serve products of this invention may be prepared by aerating a liquid mix under pressure with the aerating agent, agitating the mixture, releasing the pressurized soft-serve into a pressurized freezing zone, and discharging the final product from the freezing zone.

The liquid mixes and aerated soft-serve products of this invention generally contain up to 15% by weight butter fat, vegetable fat or animal fat. Preferably, they contain at least 2% (by weight) of the said fat. It is also

preferred that the liquid mixes and products do not contain more than 10% by weight of the said fat. A particularly preferred range for the fat, especially butterfat, content is from 6%—10% by weight. The liquid mix and the final product can also contain other well-known ingredients such as flavourings, sweeteners, malts, stabilizers and/or emulsifiers. Preferred soft-serve products are soft ice cream, frozen custard, and frozen or semi-frozen milk shakes and mousses.

The aerating agent is preferably chloropentafluoroethane or a mixture thereof with nitrous oxide. Most preferably, it is the former alone.

The amount of aerating agent present is preferably chosen such that, at 0°C., the volume of gas from the condensable aerating agent is about five times the volume of the liquid mix from which the soft-serve product is obtained, while the effective volume of aerating agent in the dispensed product at 100% overrun is about equal to the volume of the liquid mix.

In one process of obtaining the aerated products of this invention, the liquid mix is charged into a closed pressurised container and the aerating agent is then charged, most conveniently, in its liquid phase, in an amount preselected to produce a precalculated amount of overrun. The pressurized container can then be fitted directly to the freezing apparatus, but is preferably allowed to reach equilibrium and is agitated prior to such fitting to ensure aeration of the mix. After the pressurized mix container is fitted to the freezing apparatus, the mix is discharged from the container into the freezer. The pressure of the aerating agent forces the mix into the freezing zone wherein the aerating agent, distributed throughout the liquid mix, expands to a precalculated volume, aiding the cooling process. The liquid mix is forced into the freezing zone until the pressure in the zone and the container is approximately the same. By this means an aerated soft-serve product of a precalculated overrun can be obtained. The soft-serve product so prepared can then be dispensed into the atmosphere for consumption. However, if it remains in the freezer, with gentle agitation to ensure maintenance of uniform temperature throughout the soft-serve product, it maintains the preselected degree of overrun and does not degenerate or lose the desired degree of overrun, as do presently known aerated soft-serve products. Suitable apparatus for performing the process described above is disclosed in U.S. patent no. 3,004,404. This apparatus comprises two refrigerated compartments, one for refrigerating the mix composition, and the other for product freezing. The freezing chamber should contain a scraper to prevent solidification of mix along the freezer walls. The pressurized container containing mix and aerating agent is equipped with a standpipe or dip tube for dispensing the contents.

In the pressurized mix container, the aerating agent is dispersed therein, either by agitation or by handling during shipment, into small minute gaseous cells. This container can be hermetically sealed and shipped to a soft-serve product distributor ready for attachment to the soft-serve apparatus. Upon discharge of contents of the pressurized container into the freezing zone, the gaseous cells of the aerating gas expand suddenly while retaining their homogeneous consistency throughout the mix.

It has been found that with the addition of the aerating agents defined above, overruns of up to 110% can be obtained without degradation of the product. Moreover, the final product of the invention provides much less drainage and appears firmer than known such final products. The aerated products of this invention also have a smooth and homogeneous texture free of graininess.

It has also been found that when a mixture of chloropentafluoroethane and nitrous oxide is employed with frozen or semi-frozen sherbets, overrun is increased from 5—10% to about 33%. Sherbets normally contain about 2% butterfat.

Tests further indicate that the butter fat content of the mix and the final product may be lowered while maintaining the desirable effect of a rich butter fat content product, due to the presence of the aerating agent.

In order that the invention may be further understood, the following Examples are given by way of illustration only, Examples 1, 2, 4 and 6 being in accordance with the invention.

## Example 1

An 18.9 liter high-pressure tank containing about 18.1 kg. of soft ice cream mix having a butter fat content of 6% (by weight) and sufficient chloropentafluoroethane to provide a tank pressure of about 80 psig was shaken, refrigerated and attached to a soft-cream dispenser having a freezing chamber, and the tank was shaken. The mixture in the tank was then cooled to a temperature of about 1.1°C. under storage, while the freezing chamber of the apparatus was cooled to about −7.2°C. The freezing chamber had a capacity of 2.4 liters. In operation, soft ice cream was dispensed which had an overrun of about 100 per cent.

## Example 2

Vanilla-flavored ice cream mix (18 kg.) having a specific gravity of 0.92 g./cm.$^3$ and containing 10 wt.% butter fat was charged to a 19.7 liter reservoir. With the reservoir and its contents at about 4.5°C., chloropenta-

fluoroethane was admitted to a pressure of 50 psig. while shaking for six minutes, by tilting back and forth on a laboratory mixing device. After standing, 400 gms. of chloropentafluoroethane was added with shaking over a period of six minutes to ensure saturation of the mix with chloropentafluoroethane. The equilibrium pressure was 72 psig.

After standing overnight, the reservoir was attached to the dispensing machine described in Example 1 and filtered compressed air at 40—45 psig. was impressed on the reservoir via a check valve. Representative samples of dispensed product were collected and examined for overrun and drainage, with the results shown in Table I.

TABLE I

| Sample No. | Overrun[1] [%] | Temp. (°C.) | Drainage[2] (Time to First Drop) | Vol. of Drainage |
|---|---|---|---|---|
| 1 | 45 | —7.8 | N.M.** | N.M. |
| 2 | 70 | —3.9 | " | " |
| 3 | 110 | —8.9 | " | " |
| 4* | 80 | —8.9 | 25 min. | 2 cc. in 60 minutes |
| 5 | 95 | —8.9 | N.M. | N.M. |
| 6 | 95 | —8.9 | " | " |
| 7 | 90 | —7.8 | " | " |
| 8 | 85 | —7.8 | " | " |
| 9 | 90 | —7.8 | " | " |
| 10 | 85 | —6.1 | " | " |
| 11 | 85 | —6.1 | " | " |

* The reservoir and the dispenser were allowed to stand overnight between the dispensing of samples 3 and 4.

** N.M. denotes that the reading was not measured.

[1] Overrun is expressed in per cent according to the equation

$$\text{Overrun} = \frac{\text{Product Volume (cm}^3\text{)} - \text{Product wt (gms)}}{\text{Product wt (gms)}} \times 100$$

[2] Drainage was measured by placing uniform amounts of dispensed product on 10-mesh metal screens fitted in metal funnels at room temperature. The time required for the first drop to fall was noted, and the volume of drainage over a fixed time interval was recorded.

EXAMPLE 3

In order to compare the results obtained in Example 2 with results obtained from a soft serve product containing nitrous oxide, the procedure of Example 2 was carried out as follows:

The same ice cream mix as employed in Example 2 was charged (15.2 kg.) to an identical reservoir as used in Example 2. Nitrous oxide was admitted in the same manner as in Example 2 until it reached 50 psig. After standing, the pressure reached

equilibrium at 42 psig. The reservoir was then repressurized with nitrous oxide and the equilibrium pressure again fell to 42 psig., thus assuring that the ice cream mix was saturated with nitrous oxide.

The reservoir was attached to the same dispenser machine used in Example 2 and samples of dispensed product collected and examined as in Example 2, with the results shown in Table II.

TABLE II

| Sample No. | Overrun [%] | Temp. (°C.) | Drainage (Time to First Drop) | Vol. of Drainage |
|---|---|---|---|---|
| 1 | 80 | −10.0 | N.M. | N.M. |
| 2 | 70 | −6.1 | N.M. | N.M. |
| 3 | 65 | −9.5 | 3 min. | N.M. |
| 4 | 75 | −8.35 | 3 min. | 6 cc. in 10 min. |
| 5 | 80 | −8.9 | N.M. | N.M. |
| 6 | 75 | −8.9 | 2 min. | 10 cc. in 10 Min. |
| 7 | 80 | −7.2 | N.M. | N.M. |

EXAMPLE 4

3.96 kg. of the ice cream mix employed in Example 2 was placed in a 6.36 liter reservoir and pressurised as in Example 2 with 100 g. of chloropentafluoroethane. This admixture was dispensed as in Example 2 and had an overrun of about 30 per cent.

An identical sample was pressurized in the same manner with 125 g. of chloropentafluoroethane. Upon dispensing of product as described in Example 2, the results of Table III were obtained.

TABLE III

| Sample No. | Overrun [%] | Temp. (°C.) | Drainage | |
|---|---|---|---|---|
| | | | Time to First Drop (min.) | Vol, in 60 min. ($cm.^3$) |
| 1 | 110 | −7.2 | 20 | N.M. |
| 2 | 125 | −6.7 | 25 | 2 |
| 3 | 115 | −9.5 | 15 | N.M. |
| 4 | 115 | −5.5 | N.M. | N.M. |

This example shows the desirability of saturating the ice cream mix with the aerating agent.

EXAMPLE 5

A. Vanilla ice cream mix (4.09 kg. similar to the mix used in Example 2, except that it contained 6 wt.% butter-fat instead of 10%, was charged to a 6.36 liter reservoir and saturated with 50 psig. nitrous oxide for five minutes as described in Example 2. The equilibrium pressure after shaking and after standing 21 hrs. at 4.45°C. was constant at 50 psig.

Representative dispensed samples were dispensed as described in Example 2 and were examined as shown in Table IV following:

TABLE IV

| Sample No. | Overrun [%] | Time to First Drop (min.) | Drainage Vol. in 5 min. ($cm.^3$) | Drainage Vol. in 10 min. ($cm.^3$) |
|---|---|---|---|---|
| 1 | 85 | 3.5 | 2.0 | 21 |
| 2 | 95 | N.M. | N.M. | N.M. |
| 3 | 90 | 4.0 | 1.0 | 13 |
| 4 | 95 | N.M. | N.M. | N.M. |
| 5 | 90 | N.M. | N.M. | N.M. |
| 6 | 110 | N.M. | N.M. | N.M. |

B. Vanilla ice cream mixture of the same composition as in part A (4.09 kg.) was charged to a 6.36 liter reservoir and pressured with shaking with 50 psig. nitrous oxide for 30 seconds. Chloropentafluoroethane (142 g.) was injected and the reservoir was shaken for five minutes. Immediately after shaking, the pressure was 80 psig.; after standing 21 hrs. at 4.45°C. the pressure was 90 psig.

Dispensed according to the procedure of part A after standing 21 hrs., the product had good stability to drainage, as shown in Table V following:

TABLE V

| Sample No. | Overrun (%) | Time to First Drop (min.) | Drainage Vol. in 5 min. ($cm.^3$) | Drainage Vol. in 10 min. ($cm.^3$) |
|---|---|---|---|---|
| 1 | 110 | 8.5 | none | trace |
| 2 | 100 | N.M. | N.M. | N.M. |
| 3 | 100 | 7.0 | none | 0.5 |
| 4 | 90 | N.M. | N.M. | N.M. |
| 5 | 90 | N.M. | N.M. | N.M. |
| 6 | 100 | N.M. | N.M. | N.M. |

EXAMPLE 6

4.09 kg. samples of soft serve mixes were charged to 6.36 liter reservoirs. Weighed amounts of condensable aerating agents were charged to the reservoirs as shown in Table VI, column 1. Non-condensable aerating agents were supplied under pressure to the reservoirs for five minutes with shaking at the pressures shown in Table VI, column 2. The reservoirs were allowed to stand 24 hours at 4.45°C. before use. Equilibrium pressures were measured at that time and recorded in column 3 of Table VI.

Samples were dispensed as described in Example 2 and physical measurements were made as described therein with the results shown in columns 4, 5, 6 and 7 of Table VI.

TABLE VI

| Aerating Agent | 1 Wt. Condensable Aerating Agent (g.) | 2 Pressure Non-Condenable Aerating Agent (psig.) | 3 Equilibrium Pressure 24 hrs., 4.45°C. (psig.) | 4 With 6% Butterfat Mix | | | 5 With 6% Vegetable Fat Mix | | | 6 With 1—2% Butterfat (Sherbet) | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Overrun (%) | Time to 1st Drop (min.) | Vol. Collected | Overrun (%) | Time to 1st Drop (min.) | Vol. Collected | Overrun (%) | Time to 1st Drop (min.) |
| Chloropentafluoroethane | 142 | — | 75 | 48 | 2.8 | 9 cc. in 10 min. | 67 | 3.5 | 1 cc. in 10 min. | 0 | 7.7 |
| Chloropentafluoroethane/$N_2O$ | 57 | 50 | 80 | 106 | 3.0 | 1.5 cc. in 10 min. | N.M. | N.M. | N.M. | 33 | 6.0 |
| Chlorotrifluoromethane | — | 75 | 76 | 35 | 4.0 | <1 cc. in 10 min. | N.M. | N.M. | N.M. | N.M. | N.M. |

WHAT WE CLAIM IS:—

1. An aerated soft-serve product (as herein defined) having intimately dispersed therein, as aerating agent, chloropentafluoroethane, chlorotrifluoromethane, a mixture of chloropentafluoroethane and chlorotrifluoromethane, a mixture of chloropentafluoroethane and nitrous oxide, or a mixture of chlorotrifluoromethane and nitrous oxide.

2. A product according to claim 1 which contains up to 15 percent by weight butter fat, vegetable fat or animal fat.

3. A product according to claim 1 or 2 wherein the product is obtained from a soft ice cream mix, milk-shake, frozen custard mix or sherbet mix.

4. A product according to claim 2 or 3 wherein the butter fat, vegetable fat or animal fat content is at least 2 percent by weight.

5. A product according to claim 4 where-

in the butter fat, vegetable fat or animal fat content is not more than 10% by weight.

6. A product according to any of claims 1 to 5 which contains from 6 to 10 percent butter fat.

7. An aerated soft-serve product according to claim 1 substantially as herein described in any of Examples 1, 2, 4 or 6.

8. A process for preparing an aerated soft-serve product according to claim 1 which comprises aerating a liquid mix under pressure using an aerating agent chloropentafluoroethane, chlorotrifluoromethane, a mixture of chloropentafluoroethane and chlorotrifluoromethane, a mixture of chloropentafluoroethane and nitrous oxide, or a mixture of chlorotrifluoromethane and nitrous oxide, and cooling the aerated liquid mix to a temperature of between −17.7°C. and −1.1°C.

9. A process according to claim 8 wherein the mix contains up to 15 percent by weight butter fat, vegetable fat or animal fat.

10. A process according to claim 8 or 9 wherein the mix is soft ice cream mix, milkshake mix, frozen custard mix or sherbet mix.

11. A process according to claim 9 or 10 wherein the butter fat, vegetable fat or animal fat content is from 6 to 10 percent by weight.

12. A process for preparing an aerated soft-serve product according to claim 1, substantially as herein described in Example 1, 2, 4 or 6.

13. An aerated soft-serve product when produced by the process of any of claims 8 to 12.

A. A. THORNTON & CO.,
Chartered Patent Agents,
Northumberland House,
303/306 High Holborn, London, W.C.1.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1971.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.